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OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

Date: 9/19/77

Project Title: "Investigation of the Capture of Sulfur Dioxide and Oxygen by Condensation of Water in Droplets."

Project No: E-19-656

Project Director: Dr. M. J. Matteson

Sponsor: U. S. Energy Research and Development Administration

Agreement Period: From 9/1/77 Until 2/28/79 (Contract Term)

Type Agreement: Contract No. EE-77-S-05-5592

Amount: $58,094

Reports Required: Progress Report, Final Report

Sponsor Contact Person (s):

Technical Matters

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Environmental Programs
Division of Biomedical and
Environmental Research
U. S. Energy Research and
Development Administration
Washington, D. C. 20545

Contractual Matters
(thru OCA)

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Research Contracts, Procedures
and Reports Branch
Contract Division
U. S. Energy Research and
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Oak Ridge Operations, P. O. Box E
Oak Ridge, TN 37830

Defense Priority Rating: N/A

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SPONSORED PROJECT TERMINATION

Date: 7/26/79

Project Title: "Investigation of the Capture of Sulfur Dioxide and Oxygen by Condensation of Water in Droplets."

Project No: E-19-656

Project Director: Dr. M.J. Matteson

Sponsor: Department of Energy

TERMINATED

Effective Termination Date: 2/28/79

Clearance of Accounting Charges: 2/28/79

Grant/Contract Closeout Actions Remaining:

_ Final Invoice and Closing Documents
X Ann. Fiscal Report
X Ann. Report of Inventions
_ Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
_ Other _____

Assigned to: Chemical Engineering (School/Laboratory)

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CA–4 (1/79)

ORO-5592-2

INVESTIGATION OF THE CAPTURE OF SULFUR DIOXIDE AND OXYGEN BY CONDENSATION OF WATER IN DROPLETS

Progress Report

Michael J. Matteson

Georgia Institute of Technology
Atlanta, Georgia

September 1, 1977 - November 30, 1978

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY

UNDER CONTRACT NO. EE-77-S-05-5592

ABSTRACT

The first phase of this research involved the measurement of the uptake of sulfur dioxide and other gases by large, single water droplets growing by condensation or evaporating. Gases were mixed in a humid nitrogen stream and allowed to flow past a suspended cold (ca 7° C) droplet about 2.5 mm in diameter. The temperature of the droplet was monitored and the absorbed gas concentration measured after various exposure times. Gases investigated were SO_2 (1000 - 3000 ppm), NO_2 (100 - 300 ppm) and O_2 (21 - 80%). It was found in all three cases that gases absorbed during droplet growth are transferred at greater rates and in higher concentrations than the no-growth condition. If the droplets are allowed to evaporate, much less gas is absorbed; and if the droplet already contains a gas absorbed during a condensation step, some of this may be desorbed during a subsequent evaporation step. Similar tests were conducted with droplets which contained known amounts of vanadium pentoxide, manganese chloride, and carbon black. These additives all accelerated the absorption rate during condensation, however only V_2O_5 solution retained the SO_2 and prevented its desorption during evaporation.

Introduction

The absorption of gases by clouds, fogs and water droplets is a key step in the removal process for many trace gases in the atmosphere. The high acid content of rainfall in many industrial regions is attributed to the absorption of sulfur and nitrogen oxides. Upon the release of fossil fuel stack gases, containing water vapor, which are rapidly cooled from 250°F to ambient temperatures, much of the associated oxides of sulfur and nitrogen is dissolved as the water is condensed upon suspended salts and carbonaceous particles when the smoke plume is formed.

The object of the work reported here is to determine the rates of absorption of SO_2, NO_2 and oxygen at stack gas concentration by water droplets undergoing growth due to water vapor condensation. The effects on absorption of evaporation and dissolved or suspended materials are also measured.

Part I: SO_2 Absorption

This investigation was made in an effort to determine how water vapor condensation affects the uptake of SO_2 by droplets and how this absorption compares with situations where evaporation is occurring or where the water droplet is in thermal equilibrium with its surroundings.

Because of the very low concentrations of SO_2 in water, and since the sample volume is so small, measuring the amount of gas absorbed in water droplets as a function of time demanded a contact chamber where the droplets could be exposed to the desired test atmospheres for long enough exposure times to ensure that near-saturation conditions could be achieved. It was because of this requirement that we decided to use a suspended drop in a moving gas stream (Figure 1). This technique offered the advantage of being able to avoid the masking effects of mass transfer during droplet

formation. By synchronizing the droplet injection with a solenoid gas injection switch, it was possible to assure that the droplet was fully formed and internally at rest before exposure to the SO_2 stream. The water vapor concentration in the gas was regulated such that its vapor Pressure, compared to that of the water droplet at its injection temperature (supersaturation ratio or SSR) was 1.0, 1.5, 2.0, 2.5. The droplets were collected at prescribed intervals and analyzed for SO_2 according to the West-Gaeke method.

Heating of the droplet occurred because of the condensation of water vapor from the gas, the convective transfer of heat from the warmer air mass (25°C) and radiation from warmer surfaces. Droplet temperature was measured with a YSI Model 524 thermistor by suspending the droplet from both the injection capillary and the thermistor needle. These tests showed that the droplet temperature rises quite rapidly during the first 20 seconds and then slows to an equilibrium value somewhat lower than the ambient gas temperature. The water vapor condenses until the droplet vapor pressure is the same as that of the ambient gas, and then evaporation begins. Figure 2 shows the case of ambient water vapor pressure at 2.5 times that in equilibrium with a 5°C droplet. The total amount of water vapor condensed varied with SSR, and there was only evaporation taking place at an SSR = 1.0.

The results presented in Figures 3, 4, and 5 show the SO_2 absorbed by water as a function of time for various SSR and gas phase SO_2 concentrations. The results are normalized to the saturation concentration at the temperature of the droplet when sampled.

There is a definite effect on absorption as the SSR is increased above 1.0. This continues for about 15 seconds and then droplet concentration begins to decline. An explanation for the decline in SO_2 concentration is indicated

in Figure 2 which shows that condensation ceases after 15 - 20 seconds and evaporation begins. Therefore, the concentration of SO_2 is strongly connected to the condensation of water vapor. For the case of SSR = 1.0, no condensation and only evaporation occurred. This resulted in a very slow rate of absorption. A curve has been inserted showing the concentration one would expect if absorption were controlled by diffusion in the liquid phase:

$$\frac{\partial c}{\partial t} = D_{SO_2\text{-}H_2O} \quad \nabla^2 c_{SO_2}$$

The effects of dissolved salts and suspended carbon particles are presented in Figures 6, 7, and 8. Vanadium pentoxide and manganese chloride are known to catalyze the oxidation of SO_2. The cations are present in fossil fuel combustion products and their salts act as condensation nuclei upon release to the atmosphere. Soot is represented by carbon black. We chose a size distribution with a geometric mean diameter of 1.27 μm and a standard deviation (log normal) of 2.47. Soot is also present in abundance and can serve both as a nucleating agent and a catalyst for SO_2 oxidation.

In comparing SO_2 absorption during water vapor condensation with and without additives, it is apparent that all three additives accelerate the absorption rate. Although it is known that simultaneous absorption and chemical reaction will enhance mass transfer, we were interested in assessing the relative kinetics of enhanced SO_2 mass transfer due to water vapor condensation and that due to simultaneous reaction. If the chemical reaction step is too slow, then the SO_2 is released during the evaporation of the droplet. However, we notice that in the case of V_2O_5 the reaction rate is sufficiently fast to prevent the release of the SO_2 during evaporation. Therefore we have a combined absorption enhancement due to the two mechanisms.

Part II: Oxygen Absorption

See attached reprint

"Oxygen absorption in evaporating and condensing water droplets" AIHA Journal 39, 783-789 (1978). Matteson, M. J. and M. J. Oliver.

Part III: NO_2 Absorption

See attached preprint

ORO-5592-1
"Nitrogen dioxide absorption in evaporating and condensing water droplets", presented to AICHE 70th Annual Meeting, New York, November 13-17, 1977 Herrmann, J. P. and M. J. Matteson.

Part IV: Effort expended by Principal Investigator

Period September 1, 1977 - November 30, 1978

25% time

Period November 30, 1978 - February 28, 1979

25% time

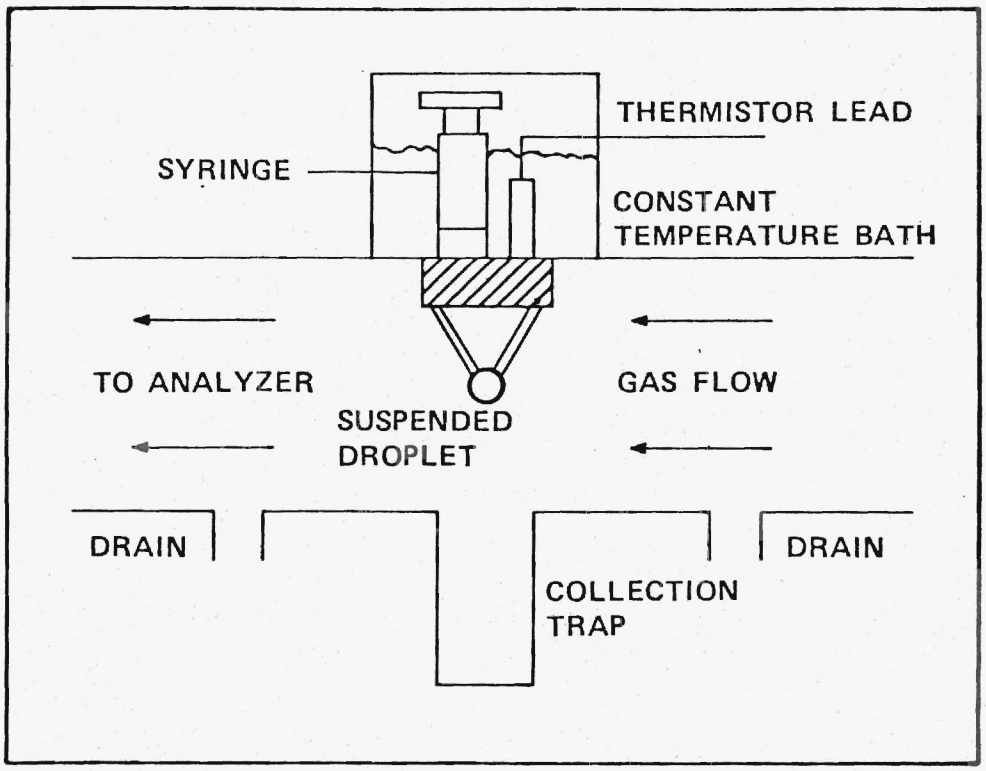


Figure 1: Contact cell

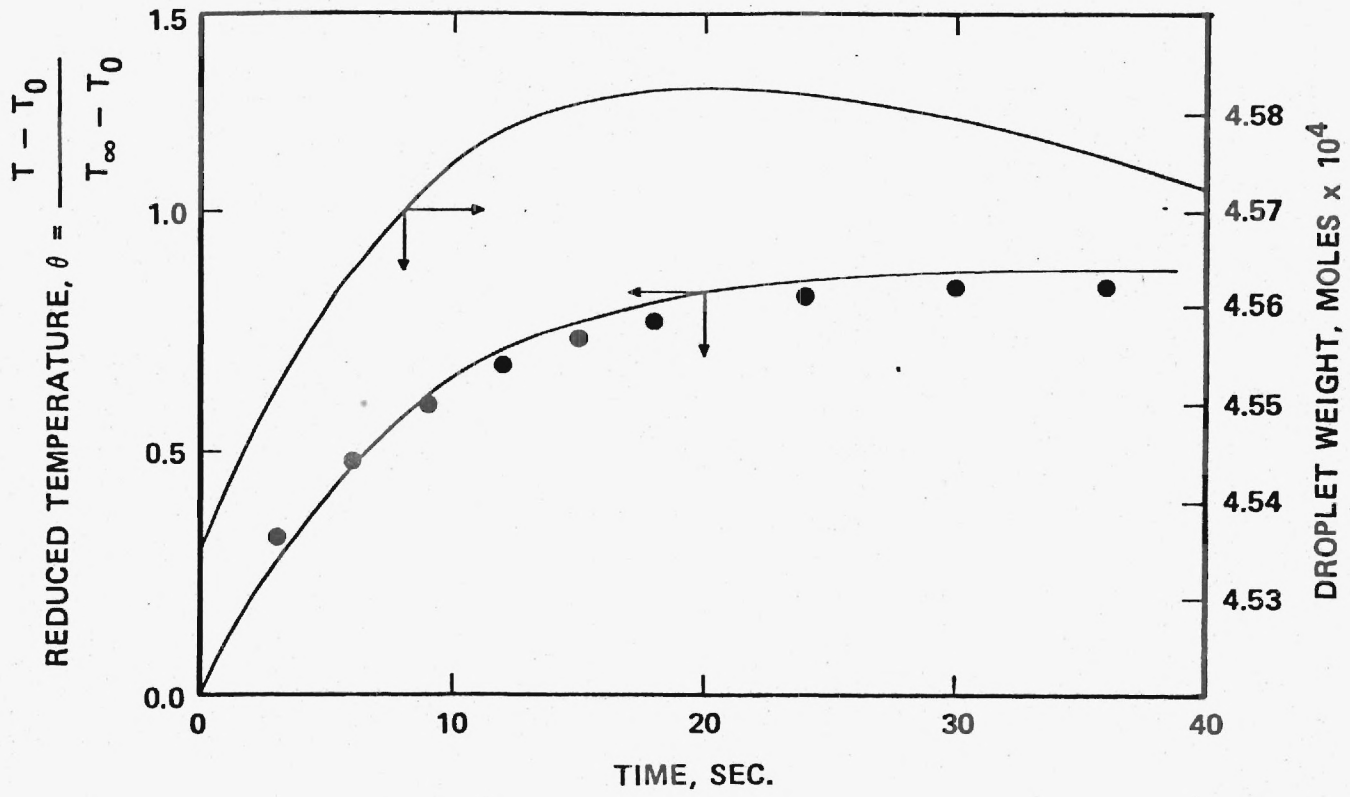


Figure 2: Droplet temperature and mass variation with time. Solid lines represent Runge-Kutta approximations based on energy-material balance for droplet with initial temperature of T_o = 5°C exposed to flowing air at T_∞ = 25°C and water vapor pressure = 2.5 times that for water at 5°C. Points are experimentally obtained values.

Figure 3: Absorption of 1000 ppm sulfur dioxide from N_2 at various water vapor condensation rates. Sulfur dioxide concentrations are normalized to saturation value at the temperature of the droplet. Dashed line represents theoretical absorption rate for droplet in equilibrium with H_2O vapor (no condensation or evaporation).

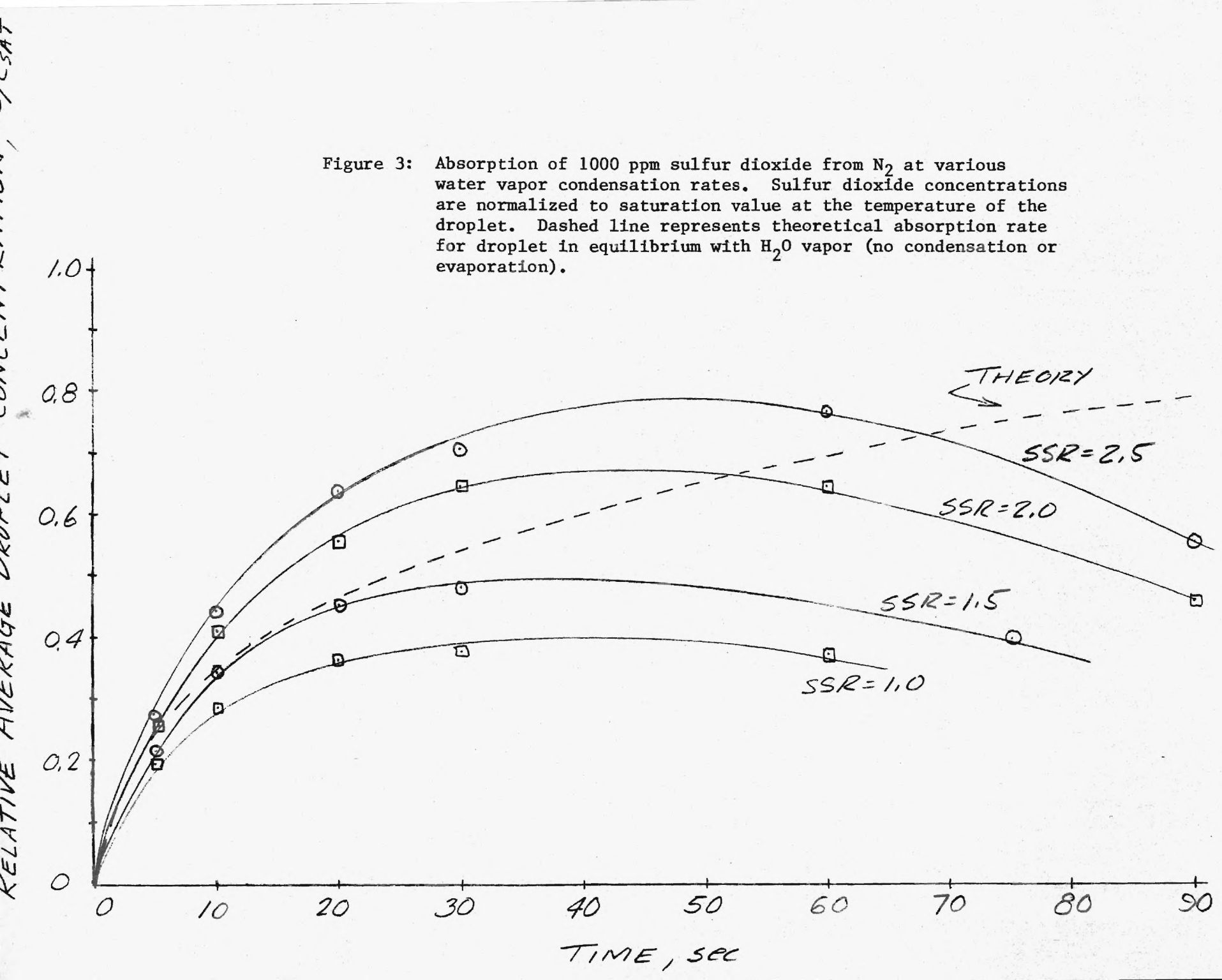


Figure 4: Absorption of 2000 ppm sulfur dioxide from N_2 at various water vapor condensation rates.

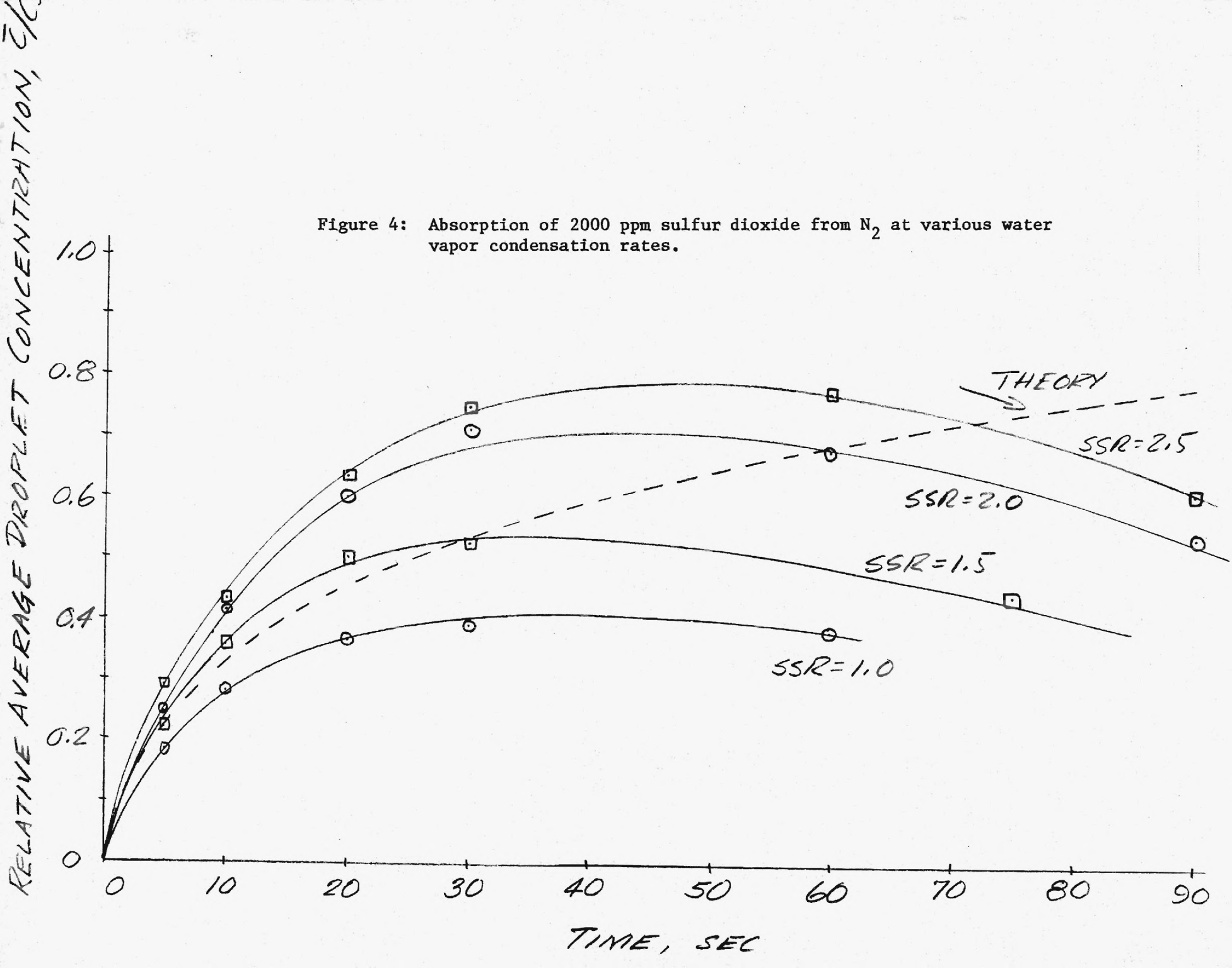
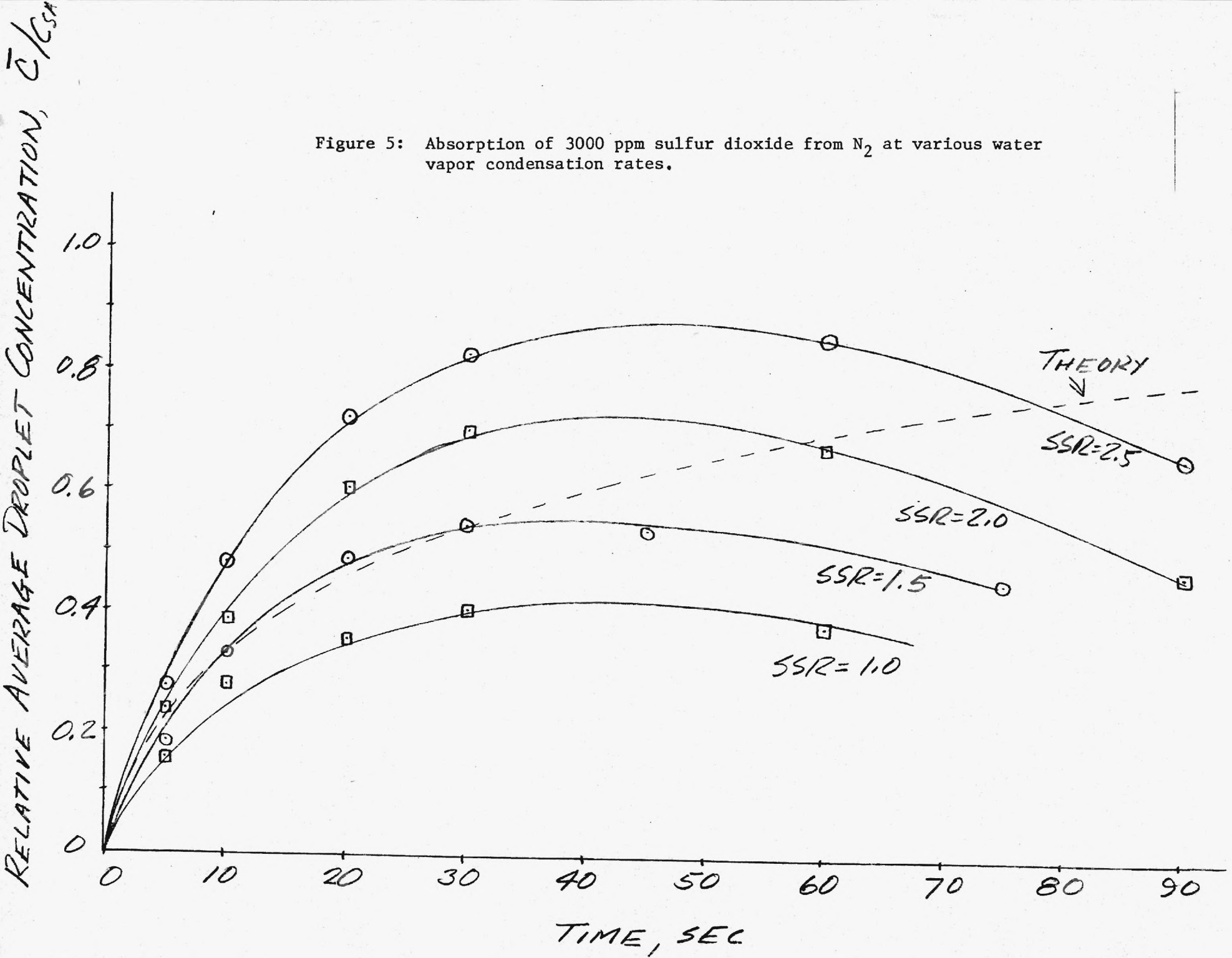


Figure 5: Absorption of 3000 ppm sulfur dioxide from N_2 at various water vapor condensation rates.



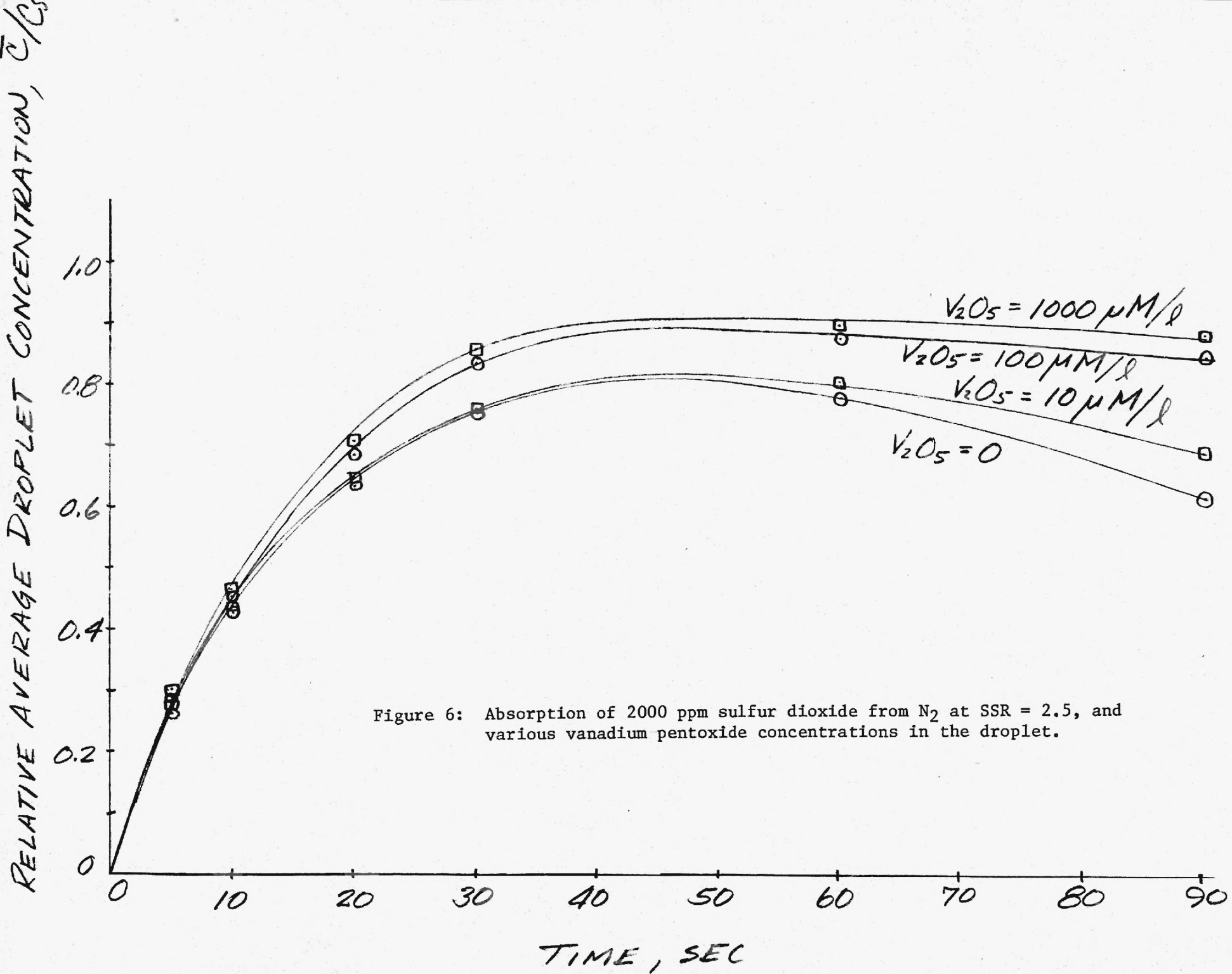


Figure 6: Absorption of 2000 ppm sulfur dioxide from N_2 at SSR = 2.5, and various vanadium pentoxide concentrations in the droplet.

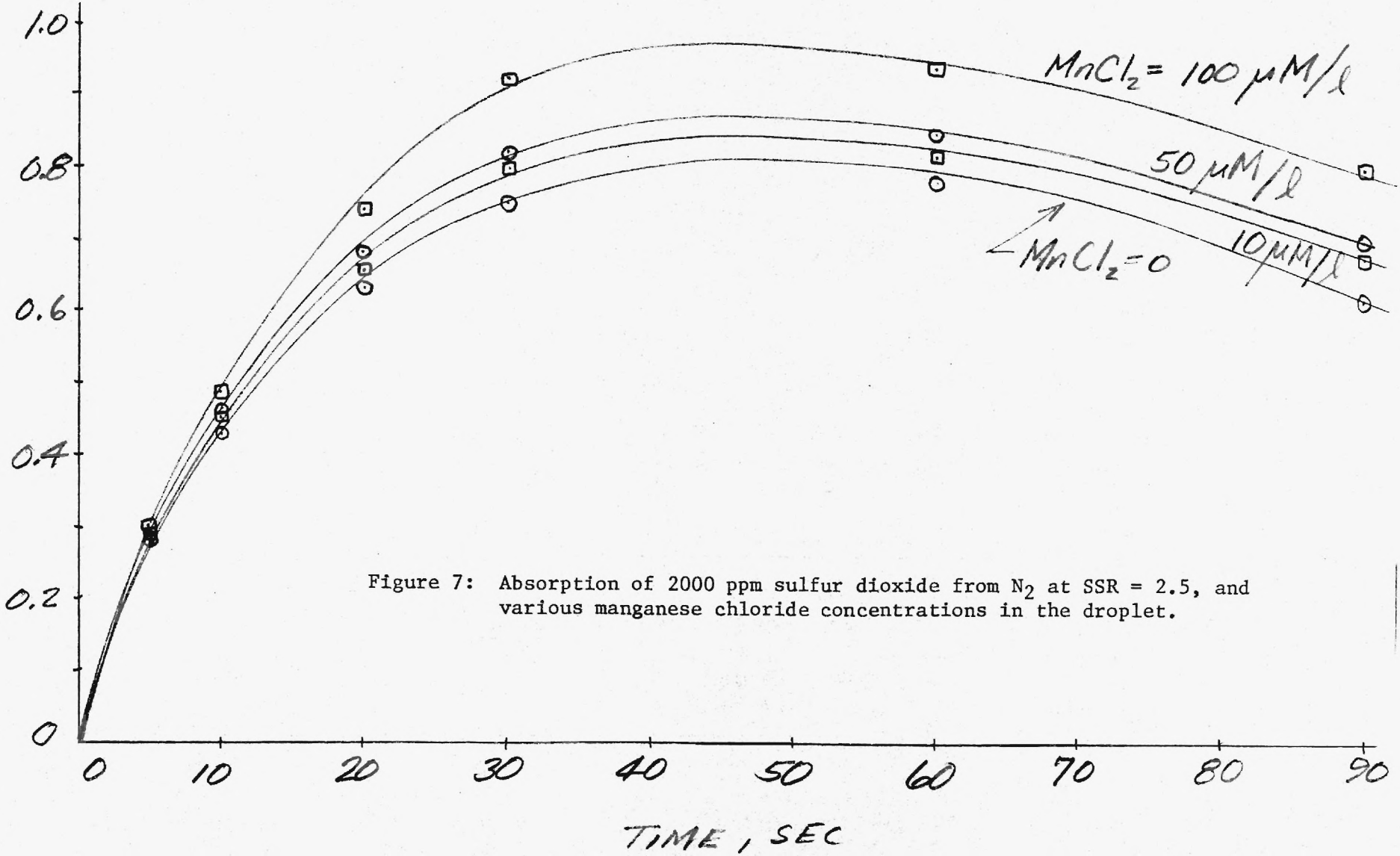


Figure 7: Absorption of 2000 ppm sulfur dioxide from N_2 at SSR = 2.5, and various manganese chloride concentrations in the droplet.

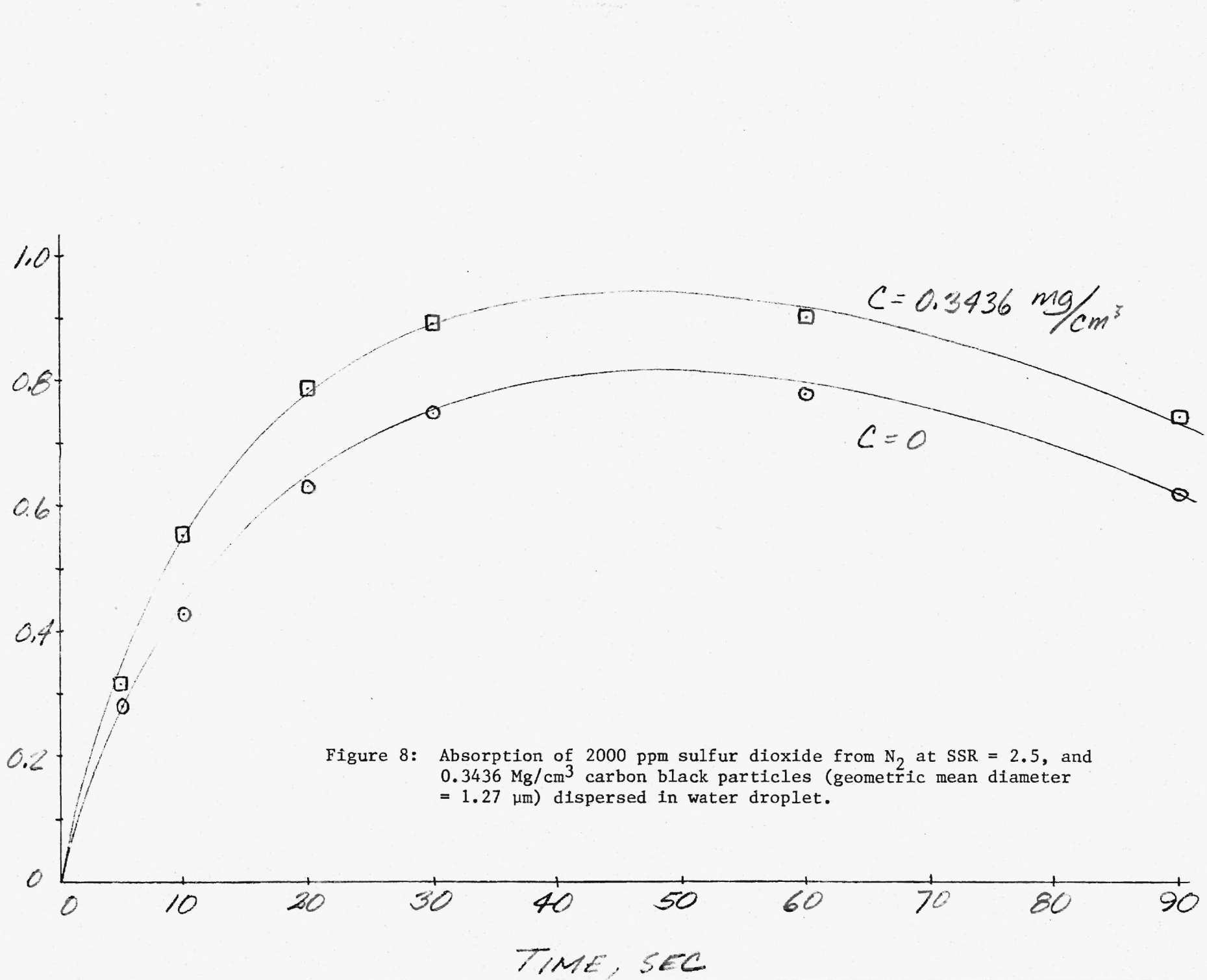


Figure 8: Absorption of 2000 ppm sulfur dioxide from N_2 at SSR = 2.5, and 0.3436 Mg/cm^3 carbon black particles (geometric mean diameter = 1.27 μm) dispersed in water droplet.

ORO-5592-1

Nitrogen Dioxide Absorption in Evaporating and Condensing Water Droplets

by

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and

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presented to

Recent Advances in Air Resource Management Symposium
American Institute of Chemical Engineers
70th Annual Meeting
New York, New York
November 13-17, 1977

Introduction

The absorption of gases by clouds, fogs and water droplets is a key step in the removal process for many trace gases in the atmosphere. The high acid content of rainfall in many industrial regions is attributed to the absorption of sulfur and nitrogen oxides. In earlier work[1] with single condensing water drops, it was demonstrated that, in the case of SO_2, when absorption occurred during droplet growth, the water actually became supersaturated with the trace gas. It is likely that the solution returns to saturation once condensation is stopped, but if dissolved salts are present, they may act to trap the gas in solution and complex it before it has a chance to be released.[2] Similar tests with oxygen as the absorbed gas demonstrated that the mechanism of enhanced mass transfer via water vapor condensation is more general and not exclusively a property of SO_2.[3]

Previous investigations of NO_2 absorption by water have been primarily concerned with fairly concentrated (>1%) amounts important in the production of nitric acid.[4] At these higher concentrations the nitrogen dioxide is predominately in the form of the dimer, nitrogen tetroxide

$$2NO_2 \rightleftharpoons N_2O_4 \qquad (1)$$

where

$K_{eq} = 3.71$ at $18.8^{o}C$.

The overall reaction with water is

$$3NO_2 + H_2O \rightleftharpoons NO + 2HNO_3 \qquad (2)$$

where

$K_{eq} = 0.137\ atm^{-1}$ at $25^{o}C$.

At higher concentrations (10%) NO_2 (N_2O_4) may actually react with water vapor in the gas phase before absorption, producing a fine mist.[5] Dekker, et al.[6] concluded that, at high concentrations, only the N_2O_4 species takes part in the formation of nitric acid.

Investigations into water absorption at low concentrations (ppm range) of nitrogen dioxide are relatively few. Borok[7] determined the Henry's Law constant for NO_2 in water to be H = 100 atm.$^{-1}$ Palmes, et al.[8] and Crecelius and Forwerg[9] found that the NO_2 is present primarily as NO_2^- with only trace amounts of NO_3 when absorbed by water in the ppm range. A possible reaction sequence for the low concentration range may be:

$$3\ NO_2 + H_2O \rightleftharpoons 2HNO_3 + NO$$

$$HNO_3 + 2\ NO + H_2O \rightleftharpoons 3\ HNO_2$$

$$HNO_2 \rightleftharpoons H^+ + NO_2^-$$

with the ionization proceeding to 100% completion as the NO_2 concentration is decreased.

Dekker concluded that the main resistance to mass transfer shifts from the gas phase to the liquid phase as the concentration is lowered. With this background it was decided to test NO_2 in the 100-300 ppm range for enhanced mass transfer via water vapor condensation.

Experimental Arrangement and Procedure

Because of the very low concentrations of NO_2 in water, and since the sample volume is so small, measuring the amount of gas absorbed in water droplets as a function of time demanded an extremely sensitive analytical procedure. We chose the Saltzman[10] method with N-(1-naphthyl) ethylene diamine dihydrochloride as the diazotization agent, which is applicable in the 0.005-5.0 ppm (water) range, and measures NO_2^- ion concentration directly.

It was further necessary to design a contact chamber where the droplets could be exposed to the desired test atmospheres for long enough exposure times to ensure that near-saturation conditions could be achieved. It was because of this requirement that we decided to use a suspended drop in a moving gas stream (Figure 1). The contact cell was a glass tube, 60 cm long and 3.5 cm inside diameter; a 24-gauge hypodermic, with tip polished and flat, was wrapped in Teflon tape, and injected at a point 40 cm from the gas entrance. A droplet 3 mm in diameter was suspended from the capillary at the axis of the tube and was exposed to a predetermined gas mixture flowing at 50.0 cm/sec. By manipulating the syringe, a droplet could be released and a new one formed when desired. By synchronizing the droplet injection with a solenoid gas injection switch, it was possible to assure that the droplet was fully formed and internally at rest before exposure to the NO_2 stream. Before beginning a series of tests droplets were sampled immediately after injection to insure that they contained no NO_2^- ions prior to exposure to the gas stream. Concentrations of NO_2 in nitrogen tested were 100 and 300 ppm; and water vapor pressures were adjusted such that the gas was super-saturated with respect to the initial droplet temperature. Supersaturation ratios (SSR) of 1.0 (no condensation), 1.5, 2.0, and 2.5 were applied at the onset of exposure. Humidity was analyzed with a Cambridge System, Model 880, Thermoelectric Dew Point Hygrometer, and nitrogen dioxide content of the gas stream was monitored with a Beckman, Model 255A UV Analyzer. Water was distilled, deionized and then passed through an oxygen removal cartridge. The syringe was immersed in a constant temperature bath at 5^oC.

Because of the necessarily long (up to 1 min) exposure times, it was not possible to maintain the droplet at a constant temperature, as was desired. Heating of the droplet occurred both because of the condensation

of water vapor from the gas and because of the convective transfer of heat from the warmer air mass ($25^{o}C$). Droplet temperature was measured with a YSI Model 524 thermistor by suspending the droplet from both the injection capillary and the thermistor needle (see Figure 1). These tests showed that the droplet temperature rises quite rapidly during the first 20 seconds and then slows to an equilibrium value somewhat lower than the ambient gas temperature. Energy and material balances were written for the droplet and these were solved using the Runge-Kutta approximation.[1] Computer solution shows that the temperature approaches the experimentally determined values quite well. The water vapor condenses until the droplet vapor pressure is the same as that of the ambient gas, and then evaporation begins. Figure 2 shows the case of ambient water vapor pressure at 2.5 times that in equilibrium with a $5^{o}C$ droplet. The total amount of water vapor condensed varied with SSR, and there was only evaporation taking place at an SSR = 1.0.

Results

The results presented in Figures 3 and 4 show the NO_2 absorbed by water as a function of time for various SSR and gas phase NO_2 concentrations. The results are normalized to the saturation concentration at the temperature of the droplet when sampled. Error bars indicate a $\pm 5\%$ variation in concentration and a ± 1 second variation in exposure time. The error in concentration is due mainly to the absorbance reading. A ± 1 second error in exposure is attributed to the sampling sequence. For the tests at both 100 and 300 ppm NO_2 in the gas phase, the droplets were practically saturated with NO_2 within 5 seconds of exposure. The dashed lines indicate the rate of NO_2 (N_2O_4) absorption according to the expression

$$N_{N_2O_4} t = (Ci)_{N_2O_4} (Dk)^{1/2} t$$

which is based on an assumed first order reaction during non-steady state diffusion into a semiinfinite medium (penetration theory).[11]

$(Ci)_{N_2O_4}$ = interfacial concentration of N_2O_4 = $(HP)_{N_2O_4}$

$H_{N_2O_4}$ = Henry's Law constant

$P_{N_2O_4}$ = partial pressure N_2O_4

$(H\sqrt{Dk})_{5^oC}$ = 8.2×10^{-5} gmoles/cm^2 atm sec (based on results of Kramers *et al.*[12]).

Because the NO_2 concentration in the gas is in the 100-300 ppm range, there should be virtually no N_2O_4 present according to the equilibrium constant expressed earlier. However, there seems to be no aqueous absorption data available in this range, so we chose to compare our results with that of Kramers *et al.*[12] for N_2O_4 and assuming $P_{N_2O_4} = P_{NO_2}$. This linear absorption rate in the first few seconds of absorption is somewhat slower than what we experienced, which one would expect, since we are dealing with a condensing system. However, the comparison helps to explain the rather rapid absorption rate in the early (0-5 seconds) stages of exposure.

There is a definite effect on supersaturation as the SSR is increased above 1.0. This continues for about 15 seconds and then droplet concentration returns to the saturation level. Tests at 60 seconds showed that the droplets remain at the saturation concentration. An explanation for the rapid decline in NO_2 supersaturation is indicated in Figure 2 which shows that condensation ceases after 15-20 seconds and evaporation begins. Therefore, the supersaturation of NO_2 is strongly connected to the condensation of water vapor. For the case of SSR = 1.0, no condensation and only evaporation occurred. As expected there is no evidence of supersaturation of NO_2. Greater supersaturation

was possible at the lower (100 ppm) than at the higher (300 ppm) NO_2 level, as was experienced in the investigations of SO_2 and O_2.[1,3]

Discussion

The rate of NO_2 absorption during water vapor condensation or evaporation is a function of the rate of diffusion coupled chemical reaction in the liquid phase, mentioned previously, and the rate of water vapor condensation or evaporation. In order to isolate the effect of the transport of water vapor on the rate of NO_2 absorption, the increase in the amount of nitrogen dioxide absorbed and the corresponding increase in the amount of water condensed for different SSR were determined for fixed values of time. These results indicate that the effect of water vapor transport on NO_2 absorption follows the relation

$$\frac{d\overline{C}_{NO_2}}{dt} = k(Y_{NO_2})^n \frac{d(H_2O)}{dt}$$

such that

$$\overline{C}_{NO_2} = k(Y_{NO_2})^n (H_2O)$$

where

Y_{NO_2} = NO_2 concentration in gas phase, mole fraction

(H_2O) = moles water condensed

$k = 3.68 \times 10^{-4}$

$n = 0.105$

Nitrogen dioxide absorption is definitely enhanced during water vapor condensation. If we compare the NO_2 concentration in the condensed water film with the value one calculates from Henry's Law:

$$\frac{\overline{C}_{NO_2}}{(H_2O)} = HY_{NO_2}$$

which, for 100 ppm NO_2 in air at 20°C, becomes

$$\frac{\overline{C}_{NO_2}}{(H_2O)} = 10^{-6} \text{ mole fr.}$$

whereas in the condensed water film we get

$$\frac{\overline{C}_{NO_2}}{(H_2O)} = 1.4 \times 10^{-4} \text{ mole fr.}$$

which represents a supersaturation by a factor of 140. The degree of supersaturation of NO_2 is diminished somewhat as the NO_2 concentration in the gas phase is increased. For instance, at 300 ppm the supersaturation factor is 52.

A possible explanation for this absorption enhancement may be that under conditions of water condensation, there is a net movement of water vapor toward the water surface, tending to drag extra NO_2 molecules along, whereas in the steady-state condition there exists an equimolar exchange of water molecules both to and from the surface. Nitrogen dioxide is absorbed in both cases, but in the non-steady state the amount of NO_2 entering the surface is enhanced somewhat by the condensing H_2O molecules. The resistance to absorption is primarily in the liquid phase, which is governed by the NO_2 concentration at the air-water interface. Therefore, if more NO_2 can be packed into the surface, this would tend to enhance the overall absorption process.

The results expressed here show clearly that, as H_2O condensation ceases, the NO_2 concentration level rapidly returns to saturation. There are several mechanisms which may promote this phenomenon: a) as evaporation

takes over the outer layers of solution are stripped away, releasing NO_2 back into the atmosphere; b) dissolved NO_2, present either as NO_2^- or NO_3^- may become too concentrated in the outer layers and revert to NO_2 and then escape as a gas; c) evaporating water molecules in the vicinity of the interface may reduce the gas phase NO_2 concentration to a level below that in the bulk gas, providing an extra driving force. The fact that the desorption is so rapid indicates that any one or a combination of the above mechanisms are acting more rapidly than the combined liquid phase diffusion-chemical reaction mechanism tending to absorb the NO_2 into the droplet interior.

The implication of these results when extended to growing fogs and clouds is that the conversion of ambient NO_2 gas to the dissolved nitrate form may proceed at a pace much faster and to much higher levels than heretofore predicted from accepted absorption theories. Dissolved salts may act to complex the ionized NO_x thereby trapping it and preventing its release during evaporation.

ACKNOWLEDGEMENT

This work was supported by Contract No. EE-77-S-05-5592 from the Department of Energy.

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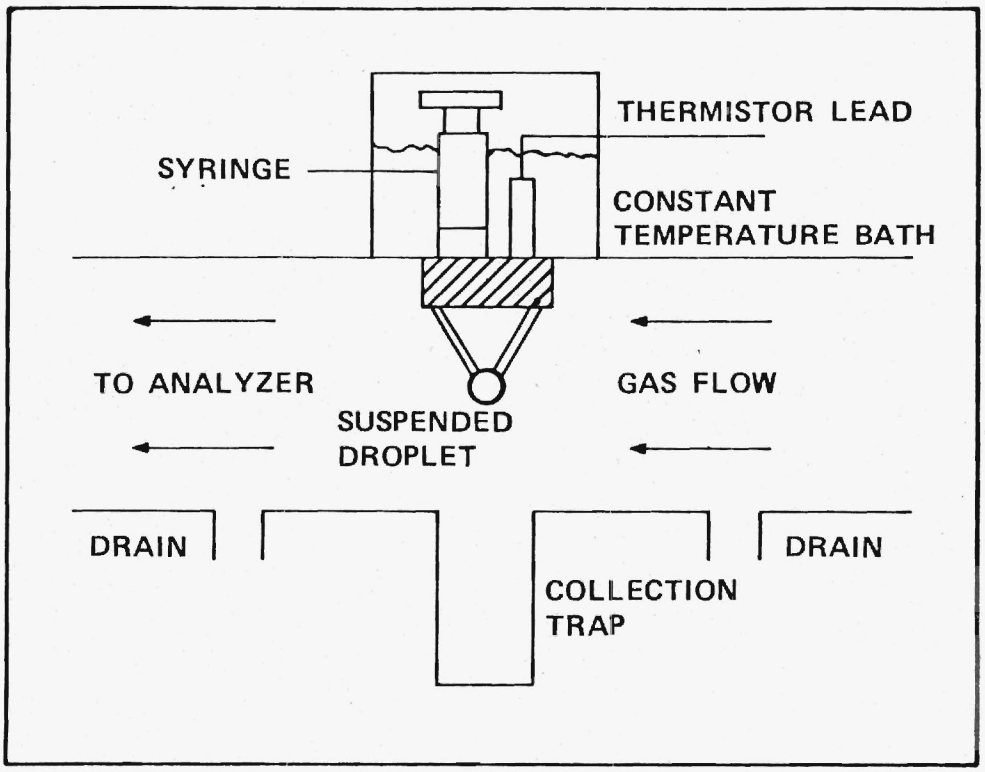
Captions

Figure 1. Contact cell

Figure 2. Droplet temperature and mass variation with time. Solid lines represent Runge-Kutta approximations[1] based on energy-material balance for droplet with initial temperature of $T_0 = 5^oC$ exposed to flowing air at $T_\infty = 25^oC$ and water vapor pressure = 2.5 times that for water at 5^oC. Points are experimentally obtained values.

Figure 3. Absorption of 100 ppm nitrogen dioxide from N_2 at various supersaturation ratios (water vapor condensation rates). Nitrogen dioxide concentrations are normalized to saturation value at the temperature of the droplet. Dashed line represents theoretical absorption rate based on N_2O_4 data from Kramers *et al*.

Figure 4. Absorption of 300 ppm nitrogen dioxide from N_2 at various supersaturation ratios (water vapor condensation rates).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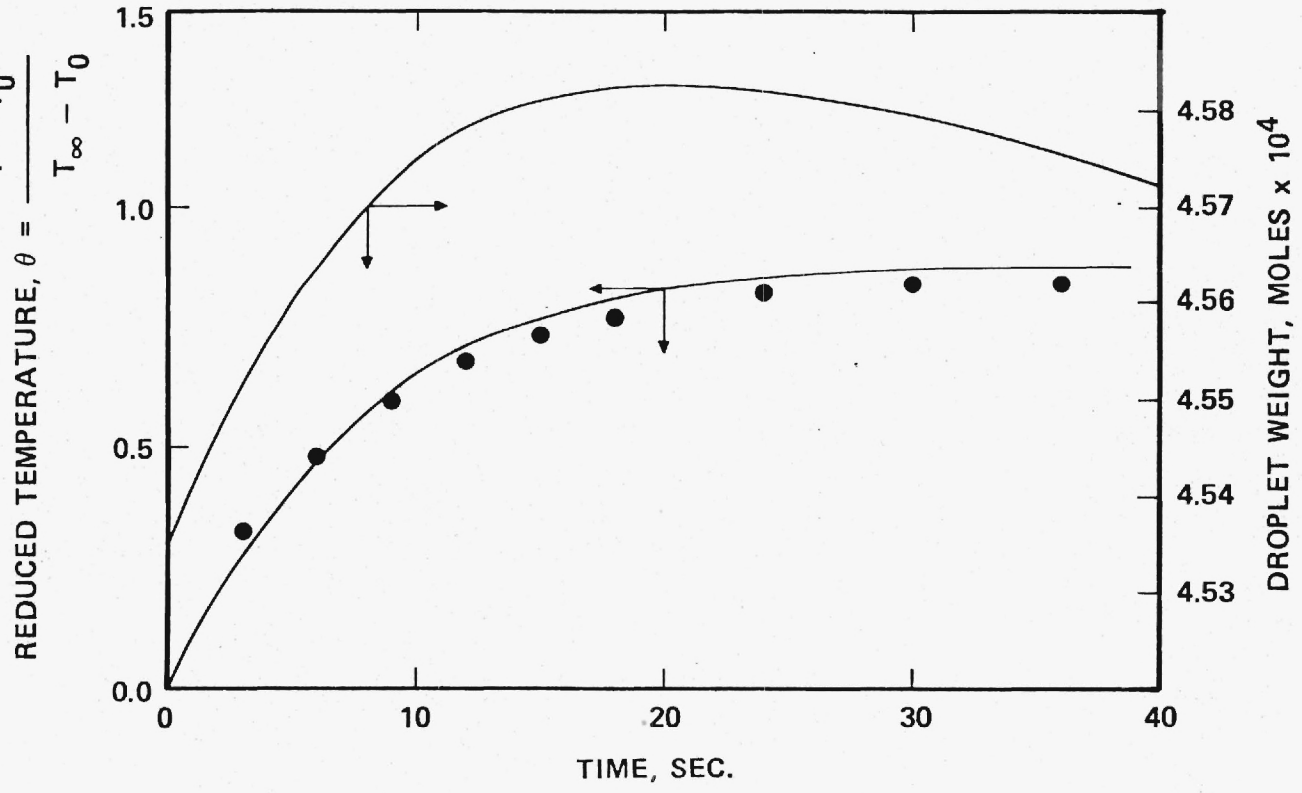
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1.0
0.5
0.0
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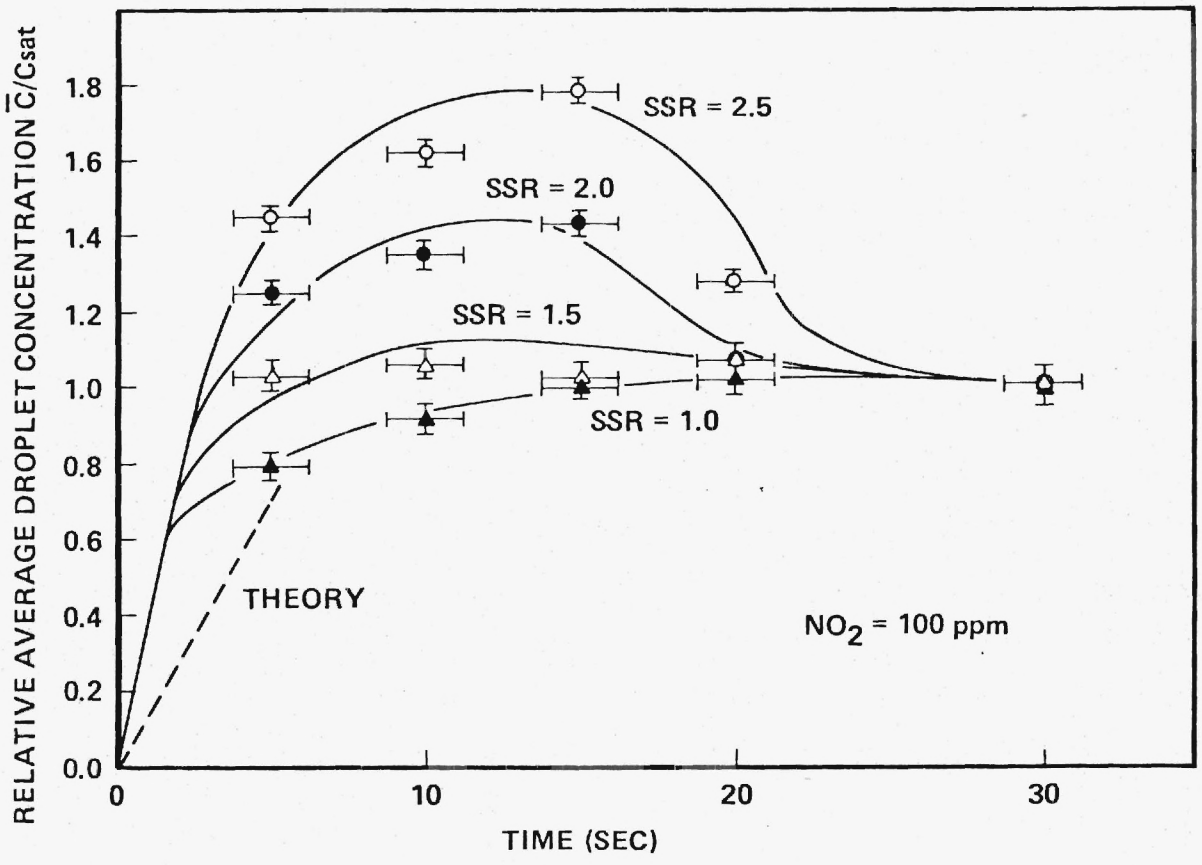
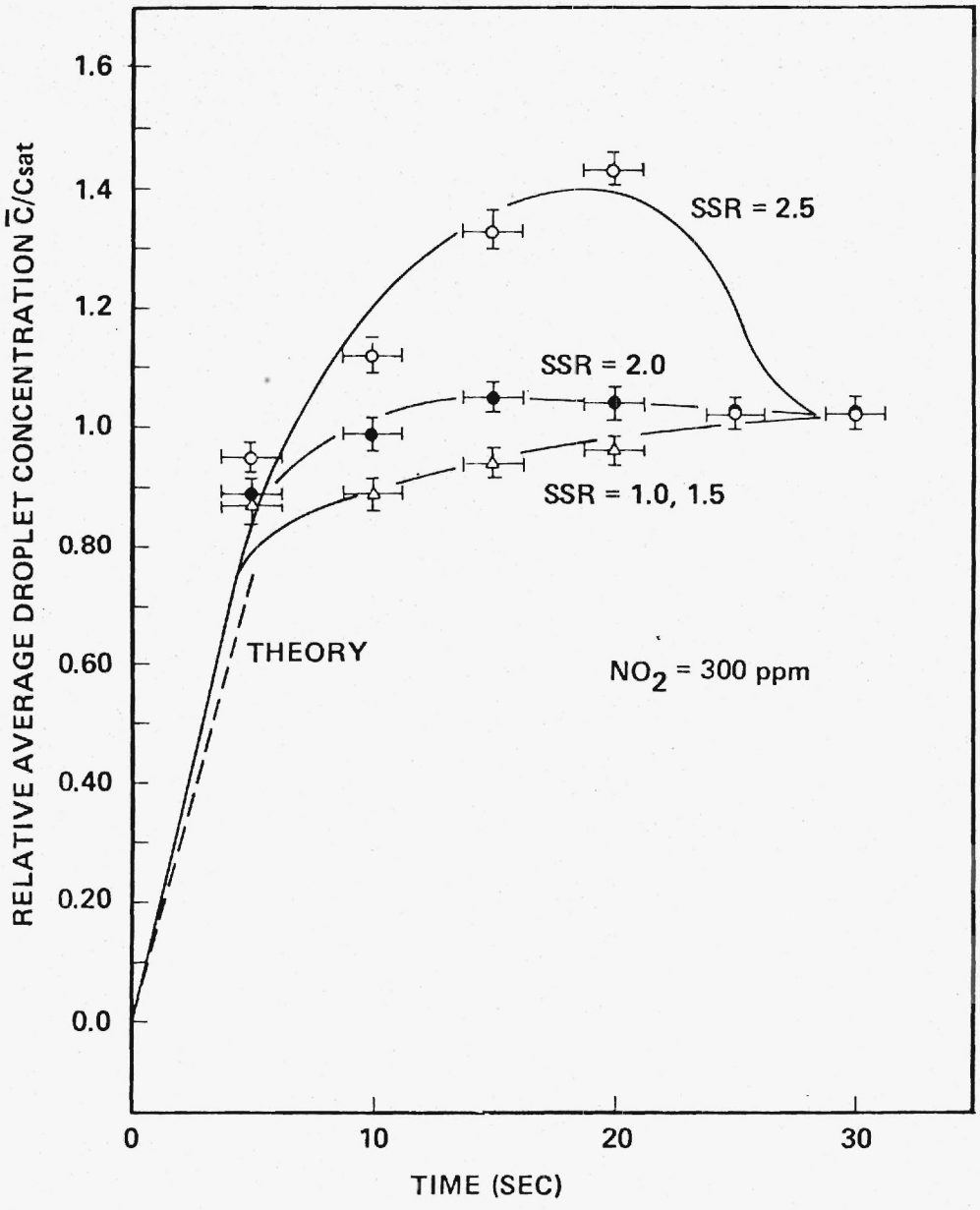
10
20
30
40
TIME, SEC.
4.58
4.57
4.56
4.55
4.54
4.53
DROPLET WEIGHT, MOLES x 10^4


RELATIVE AVERAGE DROPLET CONCENTRATION C̄/Csat
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0
10
20
30
TIME (SEC)
SSR = 2.5
SSR = 2.0
SSR = 1.5
SSR = 1.0
THEORY
NO2 = 100 ppm


RELATIVE AVERAGE DROPLET CONCENTRATION C̄/Csat
1.6
1.4
1.2
1.0
0.80
0.60
0.40
0.20
0.0
0
10
20
30
TIME (SEC)
SSR = 2.5
SSR = 2.0
SSR = 1.0, 1.5
THEORY
NO2 = 300 ppm